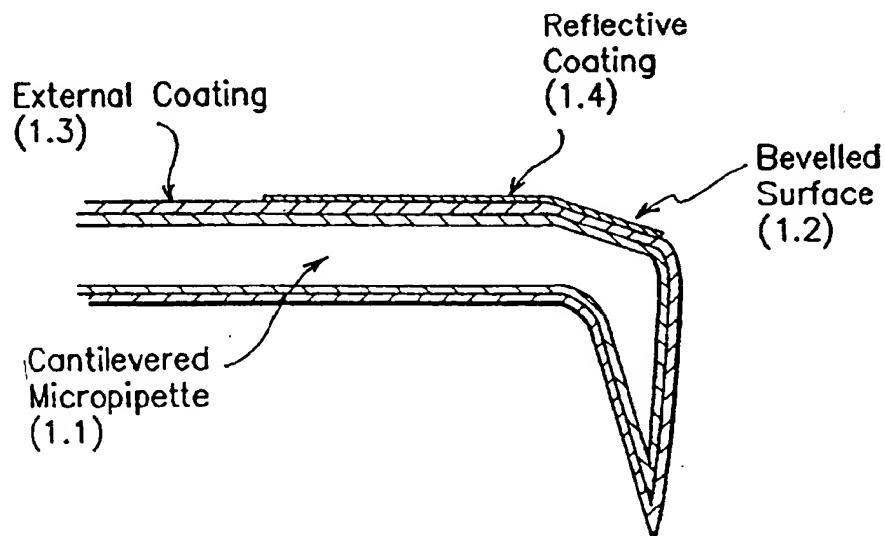




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(54) Title: PROBE MICROSCOPY



(57) Abstract

A general purpose force sensor for measuring nanometer scale surface topography and other characteristics includes a hollow micropipette (1.1) having an inner diameter of about 7.5 nanometers at its tip. The probe includes a cantilevered structure obtained by heating it near the tip to bend it. A reflective coating (1.4) is then applied to the outer surface of the micropipette.

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PROBE MICROSCOPY

Field of the Invention

The invention is a general purpose device for measuring nanometer scale surface characteristics.

5 The device integrally consists of a very sensitive force sensor for measuring surface topography and forces. The structure of the device also allows for the simultaneous monitoring of a number of other surface characteristics. In addition, the

10 device may also be used for modification and patterning with nanometer scale resolutions.

Background of the Invention

Scanned probe technologies today rely on a microscopically small tip interacting with a

15 surface as the tip is scanned in very close proximity to the surface. The interaction between the tip and the sample can typically be used both to track the surface topography and/or measure other characteristics. The two most common

20 interactions which are utilized are electron tunneling (scanning tunneling microscopy -- STM) and force sensing (scanning force microscopy -- SFM). Tunneling requires a conducting probe and a conducting sample and is thus restricted in its

25 application. Force sensing removes this restriction. Force sensing requires a structure which is sensitive enough to detect the small forces (van der walls, columbic, etc.) that are present at an interface between a tip and a

30 surface which are typically of piconewton magnitude. In addition the probe must be flexible enough so as not to deform the surface as it scans

over it. This requires a force constant on the order of 1 Newton/meter.

One of the basic requirements of a probe for practical application of scanned force sensing is 5 to have a sharp, finely tapered tip which can accurately track over and into surface corrugations. If the probe has a blunt or quickly tapered tip, scanning the tip at a constant height above the surface will not produce an accurate 10 rendition of the topography but rather a convolution of the tip structure with the surface. This is particularly significant when force imaging is to be used for metrology applications. A great deal of effort, using sophisticated 15 electron beam deposition techniques, is currently expended in order to produce sharp enough tips for such applications [Basile M.J., et al. Scanned probe tips formed by focussed ion beams, Rev. sci. instrm. 62, 2167 (1991); Kado H., Yokoyama K. 20 and Tohda T. A Novel ZnO Whisker Tip for Atomic Force Microscopy, Ultramicroscopy (1992)].

The deflection of the probe induced by the interaction with the surface force is generally 25 detected by optical means. For the detection of normal forces of the surface on the tip, the probe consists of a cantilever with a tip hanging off one end. The forces on this tip are typically measured by focusing a laser beam onto a small 30 spot on the back side of the probe. When the probe bends the small angular deviation of the beam is detected with a position sensitive detector [D. Rugar and P.K. Hansma, Phys. Today, 43, 23 (1990); K. Wickramasinghe, Sci. Am. 26, 90 (1989)]. Alternatively, the motion of the probe 35 and beam can be monitored interferometrically [D.

Rugar and P.K. Hansma, Phys. Today 43, 23 (1990); K. Wickramasinghe, Sci. Am. 26, 90 (1989)]. Both techniques require for normal force sensing a small, flat reflecting surface on which to direct 5 the beam. Alternately, lateral force sensing does not depend on a cantilevered structure.

State of Prior Art

The first normal force cantilevers were 10 fabricated by etching thin wires and mechanically bending them near the tip to produce a cantilevered structure (D. Rugar and P.K. Hansma, Phys. Today 43, 23 (1990); K. Wickramasinghe, Sci. Am. 26, 90 (1989)]. Such probes had a number of 15 problems including control over etching and the difficulty in mechanically bending the tip in a reproducible fashion. In addition such probes are not well suited to optical deflection sensing since they contain no flat region which may be used to reflect a laser beam.

20 Force cantilevers in common use today are typically microfabricated with conventional microlithography techniques [D. Rugar and P.K. Hansma, Phys. Today 43, 23 (1990); K. Wickramasinghe, Sci. Am. 26, 90 (1989)]. Such 25 probes consist of a thin silicon membrane or cantilever onto which a small sharp cone is produced [D. Rugar and P.K. Hansma, Phys. Today 43, 23 (1990); K. Wickramasinghe, Sci. Am. 26, 90 (1989)]. At the tip of the cone an additional 30 filament is often grown to produce a sharper and finer tapered tip. The mechanical characteristics of such probes are determined by the materials used, tip mass and geometry. Typical force constants for such tips are in the 0.1 to 10

Newton/Meter range. These probes, however, are not very suitable for other forms of scanned probe microscopy.

Summary of the Invention

5 The invention is a method and a device for producing a general purpose probe for all forms of scanned probe imaging and patterning. The structures produced with this method are immediately compatible with all the force
10 deflection sensing techniques in use today.

Description of the Invention

15 The device is based on a glass or quartz micropipette or fiber which can be pulled down to a variety of dimensions at the tip with 10 nanometers the smallest outer diameter achieved thus far. The micropipettes remain hollow and can have an inner diameter at the tip of 7.5
20 nanometers. These probes may be pulled with a very gradual taper giving a cone angle at the tip of only a few degrees, or can be tapered with larger cone angles if the application so requires.

25 For lateral force sensing nothing further needs to be done. For the addition of normal force sensing the probe is then given a cantilevered structure by locally heating it near the tip and applying a small force to bend the tip when the glass or quartz becomes soft. This is shown schematically in Figure 1. Localized heating is achieved by focusing a CO laser to a
30 small spot near the tip. A stream of air is directed at the tip region while the heating is taking place. This serves two purposes. First, the air cools the tip so that there will not be

excessive heat conduction in the glass which could melt the tip of the probe and second, the air flow provides sufficient force to bend the very tip as soon as the glass or quartz becomes sufficiently soft. The bend radius is determined principally by the size of the laser heating spot. Focusing a CO₂ laser down to a diffraction limited 10 μ spot can readily produce bends a few 10's of microns from the tip.

Once the cantilevered structure is obtained the crucial polishing step takes place to provide the incorporation of optical deflection sensing techniques. This is done by inserting the cantilevered structure into a micromanipulator and bringing the end of the bent region into contact with a rotating polishing surface as shown in Figure 2. The polished region may be as small as several microns in diameter. This produces an optically flat surface in which a laser beam may be reflected off to monitor the deflection of the cantilever. This polished region is just above the bent section of the tip for maximum deflection sensitivity.

After the tip is bent a reflecting metallic coating may be deposited on the polished section to enhance the reflectivity. A metal coating may be further applied to the entire outer region of a micropipette probe and the walls of a fiber probe to produce a structure which may also be used as a near-field subwavelength point of light.

Such bent polished structures can simultaneously be used to pass light through the device to the tip which, as noted above, can be transformed into a near-field aperture by an appropriate metal coating. With bent fibers light

is guided around the bend. With pipettes a high index liquid can be made to fill the pipette void and this also permits the transmission of light around the bend and through the near-field, sub-wavelength aperture at the tip. The resulting sub-wavelength point of light can be used for imaging and patterning while force is used, either normal or lateral, to monitor the topography and force characteristics of the surface.

5 Furthermore, in such a structure the metallic coating at the tip can be used to measure simultaneously tunneling currents to determine the tunneling characteristics. With micropipettes these same structures can be readily filled with

10 an optically or electrically excited light-emitting substance to produce a sub-wavelength source of light with many of the above sensing capabilities. The light emitting substance at the tip of the pipette can also be used to monitor

15 specific ions or sense surface charge.

20 Alternately, micropipette structures can be produced with a metal wire down the hollow interior extending to the tip. The coating of metal on the outside of this structure if it is generated using a different metal from that in the

25 interior, permits the production of a thermal sensor with the force and tunneling characteristics noted above. Such a metal sandwiched glass structure with a transparent

30 glass tip could also be used to propagate light without evanescent losses in the subwavelength region. This structure would be the optical analog to an electrical coax. As another alternative sol-gel conducting glass can also be

35 deposited in the tip and this glass can be

embedded with optically excited materials to produce a structure which could monitor optical, electrical and the conductive nature of surfaces. The essence of all these structures are the 5 multichannel sensing capabilities which have been patently absent in scanned probe microscopes because no elements such as the ones noted here have been devised.

Parts of the Device

10 A schematic of a device produced according to the present invention is shown in Figure 3. It consists of the following parts:

1. A glass or quartz micropipette tapered to produce a hole at the tip that can be as small as 15 7.5 nm (part number 1.1),

or

20 A glass or quartz optical fiber tapered to produce a tip outer diameter that can be as small as 10 nanometers (part number 1.1), which can be bent, for normal force sensing, near the tip to produce a cantilevered structure and polished just above the bend to produce an optically flat surface (part number 1.2).

25 2. A material (part number 1.3) such as aluminum or gold optionally deposited along the outer surface of the probe to provide for an opaque coating if required.

30 3. A material (part number 1.4) such as aluminum deposited on the beveled, polished surface of part 1.1 to provide a reflecting surface.

4. A material (part number 1.5) optionally inserted into the very tip of the micropipette probe which acts as a specific chemical, optical or thermal sensor for various local phenomenon.

5 5. The probe is incorporated into a micropositioning instrument.

6. The bent probe can be inserted with its micropositioner under the lens of a regular microscope which can be used together with an 10 interferometric measurement through this lens to sense the deflection the micropipette cantilever with the lens also being used for collection of light from the sample and illumination of the sample.

15 **Operation of the Device**

The probe is brought into the near field of the surface by either monitoring lateral force in a non-bent structure or by monitoring the deflection of the cantilever in a bent structure 20 by normal force impinging on the tip of the bent pipette or fiber. Then the structure is scanned along the surface either in contact or in near-contact by monitoring surface forces while the other attributes of the tip are used to monitor 25 simultaneously the chemical, optical, electrical or thermal characteristics of the surface.

Advantages Over Prior Art

The probes produced with the technique described here have a number of advantages over 30 presently available scanned force probes. First, the initial pulling technique allows for simply

and accurately controlling and adjusting the force characteristics and constants of the probe itself. Second, the pulling process naturally produces very sharp and finely tapered tips which are 5 required for accurate force imaging. Conventionally produced tips require a complex and poorly understood growth of a fine filament, with the aid of electron beam deposition techniques, at the tip of the microfabricated cone as an 10 additional step after the sensor is completed. Finally the micropipette and fiber probes allow numerous other surface characteristics to be monitored simultaneously with force sensing. This 15 includes near-field optical interactions and, in the case of the micropipette probe a variety of specific sensors, may be placed within the tip.

Claims

1. A device consisting of a structure such as a tapered micropipette or optical fiber drawn to a tip diameter which can be as small as 10 nanometers or less and which can be used as a lateral force sensor while being simultaneously used to produce a point source of subwavelength light by coating the outside of this structure with a metal such as aluminum, and passing light through the subwavelength tip or producing light at the subwavelength tip with appropriate materials that can be optically or electrically excited so that this light can be used for near-field scanning optical microscopy and lithography.
2. A device consisting of a structure such as a tapered micropipette drawn to an inner tip diameter which can be as small as 10 nanometers or less which is bent near the tip to produce a cantilevered structure suitable for normal force sensing and polished just above the bend to produce an optically flat surface for monitoring the deflection of the cantilever.
3. A device consisting of a structure as in claim 2 which may be coated along the outer walls to produce a probe suitable for near-field scanning optical microscopy and lithography.
4. A device consisting of a structure as in claim 3 in which a material is inserted into the tip which acts as a specific chemical, spectroscopic, surface charge or other sensor to the local environment.
5. A device consisting of a structure as in claim 4 in which a metal is the material inserted into the inner void of the micropipette and an appropriate different metal is chosen to coat the

micropipette on the outside such that a highly localized thermocouple is generated at the tip.

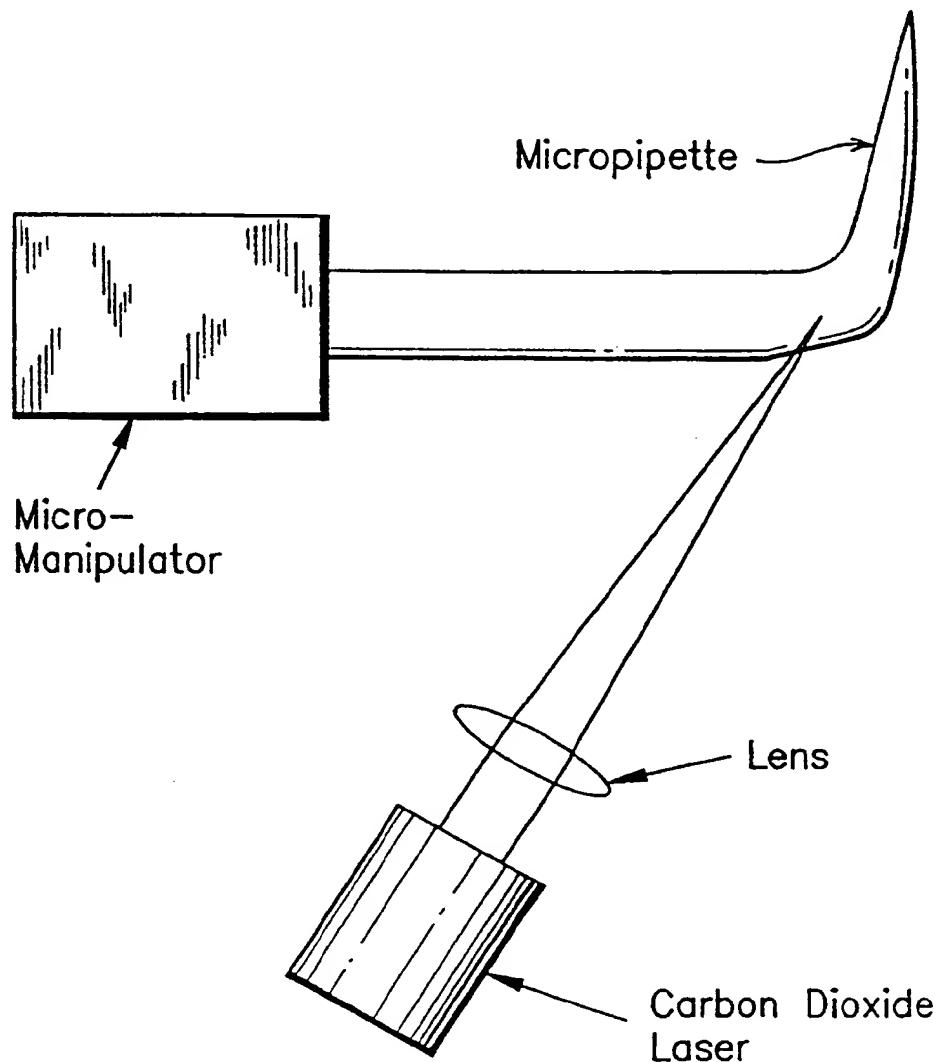
6. A device consisting of a structure such as a tapered optical fiber drawn to a tip outer diameter as small as 10 nanometers or less which is bent near the tip to produce a cantilevered structure suitable for force sensing and polished just above the bend to produce an optically flat surface for monitoring the deflection of the cantilever.

7. A device which incorporates the bent probe into a micropositioning instrument which can then be inserted with its micropositioner under the lens of a regular microscope which can be used together with an interferometric measurement through this lens to sense the deflection of the micropipette cantilever with the same lens and also while using this lens for collection of light from the sample and illumination of the sample.

8. A method for producing ultrafine cantilevered glass or quartz micropipette or optical fiber probes with force sensing characteristics by drawing a micropipette or fiber to dimensions as small as 10 nm or less and then bending the tip from a few tens of microns from the tapered end with a focused carbon dioxide laser beam.

9. A method for producing an optically flat reflecting surface just above the bend of a cantilevered micropipette or fiber probe to allow optical deflection sensing technology to be used to monitor the cantilever bending by holding the micropipette or fiber appropriately in a micromanipulator and using a polishing wheel to polish the outer surface of the micropipette or fiber.

1/2



Tip bends away from heat

FIG. 1

SUBSTITUTE SHEET (RULE 26)

2/2

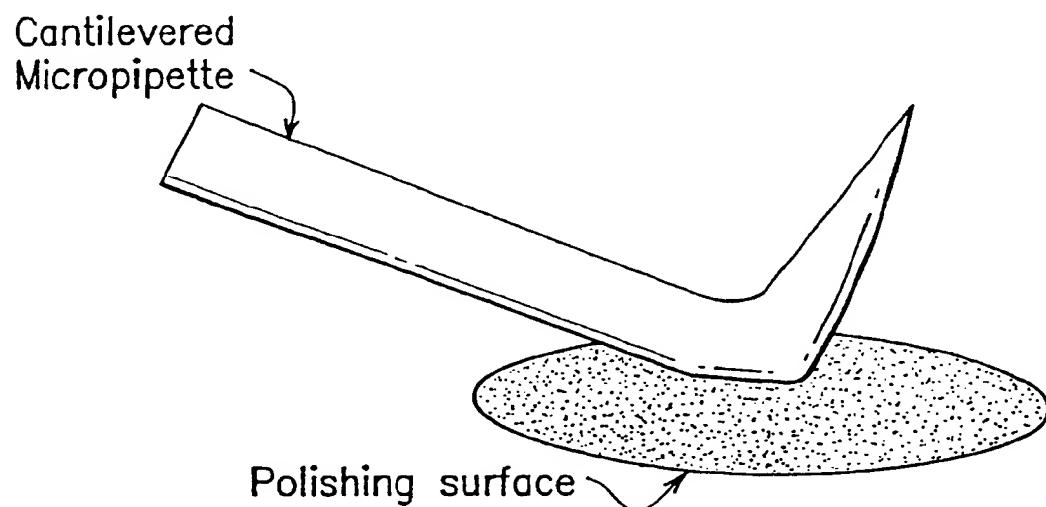


FIG. 2

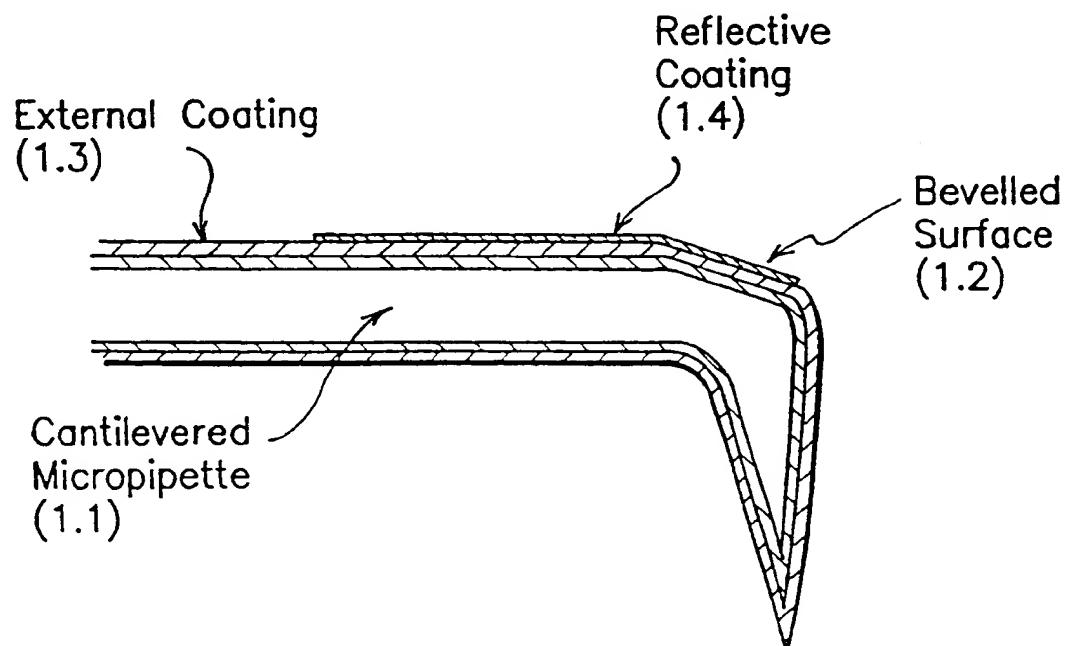


FIG. 3

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/08691

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : H01J 37/00

US CL : 250/306, 307

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 250/306, 307, 234, 216

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y,P	US, A, 5,264,698 (KOPELMAN ET AL.) 23 November 1993. See entire document.	1-7, 9
Y,P	US, A, 5,289,004 (OKADA ET AL.) 22 February 1994. See entire document.	1-7, 9
Y,P	US, A, 5,304,795 (FUJIHIRA ET AL.) 19 April 1994. See Fig. 5.	1-7, 9
A,P	US, A, 5,333,495 (YAMAGUCHI ET AL.) 02 AUGUST 1994. . See entire document.	
A	US, A, 5,105,305 (BETZIG ET AL.) 14 April 1992. See entire document.	
A	US, A, 4,917,462 (LEWIS ET AL.) 17 April 1990. See entire document.	

Further documents are listed in the continuation of Box C.

See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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